

Supporting Information Available.

Methyl 4,6-O-benzylidene-2,3-dideoxy-3-C-formyl- α -D-erythro-hex-2-enopyranoside (**1**): white crystals, mp. 149-150 °C (hexane-ethyl acetate, lit.:¹ 155-158 °C). $[\alpha]_D^{27} = +17.6$ (c 0.8, CHCl₃). IR (KBr): ν_{\max} (cm⁻¹) = 2976, 2890, 1690 (CO aldehyde), 1452, 1388, 1318, 1122, 1084, 964, 756. ¹H NMR (CDCl₃, 200 MHz): $\delta = 9.67$ (s, 1H, H aldehyde), 7.50 (m, 2H, aromatic), 7.37 (m, 3H, aromatic), 6.58 (t, J = 2.5 Hz, 1H, vinyl), 5.69 (s, 1H, benzylic), 5.08 (dd, J₁ = 2.5 Hz, J₂ = 1.3 Hz, 1H, anomeric), 4.50 (m, 1H, C-4 H), 4.37 (m, 1H, C-6 H_{eq}), 3.91 (m, 2H, C-5 H and C-6 H_{ax}), 3.51 (s, 3H, OCH₃). ¹³C NMR (CDCl₃, 50 MHz): $\delta = 189.2$ (C-carbonyl), 139.6 (C-3), 138.9 (C-2), 137.0 (C-aromatic), 128.9 (C-aromatic), 128.1 (2C, aromatic), 126.0 (2C, aromatic), 101.8 (C-7), 95.5 (C-1), 74.0 (C-4), 69.0 (C-6), 63.4 (C-5), 56.3 (OCH₃). HRMS: M⁺ m/e obs.: 276,0992 (C₁₅H₁₆O₅ m/e calc.: 276,1002). LRMS (E.I.): m/e (rel. int.): 276 (48), 245 (10), 149 (57), 127 (100), 105 (31), 99 (12), 91 (37), 77 (20).

Methyl 3-acetyl-4,6-O-benzylidene-2,3-dideoxy- α -D-erythro-hex-2-enopyranoside (**3**): white crystals, mp. 169-170 °C (hexane-ethyl acetate). $[\alpha]_D^{21} = +31.9$ (c 1.025, CHCl₃). IR (KBr): ν_{\max} (cm⁻¹) = 2952, 2917, 1696 (CO ketone), 1641, 1450, 1388, 1364, 1261, 1248, 1084, 801, 738, 696. ¹H NMR (CDCl₃, 200 MHz): $\delta = 7.46$ (m, 2H, aromatic), 7.36 (m, 3H, aromatic), 6.43 (dd, J₁ = 2.8 Hz, J₂ = 2.4 Hz, 1H, vinyl), 5.68 (s, 1H, benzylic), 5.01 (dd, J₁ = 2.6 Hz, J₂ = 1.3 Hz, 1H, anomeric), 4.48 (td, J₁ = 8.5 Hz, J₂ = 2.0 Hz, J₃ = 1.5 Hz, 1H, C-4 H), 4.38 (complex m, 1H, C-5 H), 3.92 (complex m, 2H, C-6 H_{eq} and C-6 H_{ax}), 3.47 (s, 3H, OCH₃), 2.34 (s, 3H, CH₃). ¹³C NMR (CDCl₃, 50 MHz): $\delta = 196.6$ (C-carbonyl), 140.9 (C-3), 137.1 (C-aromatic), 132.0 (C-2), 128.9 (C-aromatic), 128.1 (2C, aromatic), 126.0 (2C, aromatic), 101.8 (C-7), 95.6 (C-1), 74.7 (C-4), 69.0 (C-6), 63.6 (C-5), 56.2 (OCH₃), 28.3 (CH₃). HRMS: M⁺ m/e obs.: 290,1154 (C₁₆H₁₈O₅ m/e calc.: 290,1159). LRMS (E.I.): m/e (rel. int.): 290 (46), 259 (9), 213 (8), 149 (100), 105 (28), 91 (37), 77 (26), 55 (8), 43 (64).

Methyl 4,6-O-benzylidene-3-cyano-2,3-dideoxy- α -D-erythro-3-propionyl-hex-2-enopyranoside (**8**): white crystals, mp. 187-188 °C (hexane-ethyl acetate). $[\alpha]_D^{25} = +26.9$ (c 0.68, CHCl₃). IR (KBr): ν_{\max} (cm⁻¹) = 2926, 1702 (CO, ketone), 1643, 1453, 1387, 1365, 1204, 1087, 1014, 681. ¹H NMR (CDCl₃, 200 MHz): $\delta = 7.46$ (m, 2H, aromatic), 7.35 (m, 3H, aromatic), 6.38 (dd, J₁ = 2.7 Hz, J₂ = 2.3 Hz, 1H, vinyl), 5.67 (s, 1H, benzylic), 5.00 (m, 1H, anomeric), 4.51 (m, 1H, C-4 H), 4.34 (m, 1H, C-5 H), 3.89 (m, 2H, C-6 H_{eq} and C-6 H_{ax}), 3.48 (s, 3H, OCH₃), 2.66 (m, 2H, CH₂), 1.08 (t, J = 7.2 Hz, 3H, CH₃). ¹³C NMR (CDCl₃, 50 MHz): $\delta = 199.6$ (C-carbonyl), 140.7 (C-3), 137.0 (C-aromatic), 131.0 (C-2),

128.8 (C-aromatic), 128.0 (2C, aromatic), 125.9 (2C, aromatic), 101.7 (C-7), 95.5 (C-1), 74.6 (C-4), 68.9 (C-6), 63.5 (C-5), 56.1 (OCH₃), 33.7 (CH₂), 7.4 (CH₃). HRMS: M⁺ m/e obs.: 304,1302 (C₁₇H₂₀O₅ m/e calc.: 304,1315). LRMS (E.I.): m/e (rel. int.): 304 (28), 149 (100), 105 (24), 91 (31), 77 (14), 57 (52), 43 (8).

Methyl 4,6-O-benzylidene-3-cyano-2,3-dideoxy- α -D-erythro-hex-2-enopyranoside (**9**): white crystals, mp. 214.0-215.0 °C (benzene/hexane; lit.:² 214.5-215.5 °C). $[\alpha]_D^{28} = +60.7$ (c 0.94, CHCl₃). IR (KBr): ν_{\max} (cm⁻¹) = 2982, 2918, 2227 (CN), 1453, 1388, 1362, 1186, 1121, 1087, 1058, 988, 763, 697. ¹H NMR (CDCl₃, 200 MHz): $\delta = 7.53$ (m, 2H, aromatic), 7.40 (m, 3H, aromatic), 6.50 (t, J = 2.5 Hz, 1H, vinyl), 5.62 (s, 1H, benzylic), 5.00 (dd, J₁ = 2.4 Hz, J₂ = 1.5 Hz, 1H, anomeric), 4.33 (dd, J₁ = 9.2, J₂ = 3.4 Hz, 1H, C-6 H_{eq}), 4.18 (m, 1H, C-4 H), 3.85 (m, 2H, C-5 H and C-6 H_{ax}), 3.47 (s, 3H, OCH₃). ¹³C NMR (CDCl₃, 50 MHz): $\delta = 140.6$ (C-2), 136.3 (C-aromatic), 129.1 (C-aromatic), 128.1 (2C, aromatic), 126.0 (2C, aromatic), 117.0 (CN), 113.7 (C-3), 101.9 (C-7), 94.7 (C-1), 72.8 (C-4), 68.4 (C-6), 63.6 (C-5), 56.4 (OCH₃). HRMS: M⁺ m/e obs.: 273,1000 (C₁₅H₁₅O₄N m/e calc.: 273,1004). LRMS (E.I.): m/e (rel. int.): 273 (45), 242 (13), 149 (36), 124 (100), 105 (30), 91 (21), 77 (17), 69 (15), 60 (11), 45 (17).

Methyl 3-cyano-2,3-dideoxy- α -D-erythro-hex-2-enopyranoside (**10**): white crystals, mp. 129-130 °C (hexane-ethyl acetate). IR (KBr): ν_{\max} (cm⁻¹) = 3460 (OH), 3380, 2916, 2236 (CN), 1452, 1385, 1084, 1062, 988, 966, 762, 701. ¹H NMR (CDCl₃, 200 MHz): $\delta = 6.51$ (dd, J₁ = 3.0 Hz, J₂ = 1.9 Hz, 1H, vinyl), 4.99 (bd, J = 2.3 Hz, 1H, anomeric), 4.36 (bt, J = 6.8 Hz, 1H, C-4 H), 3.88 (bt, J = 4.4 Hz, 2H, C-6 H), 3.78 (dt, J₁ = 9.1 Hz, J₂ = 3.9 Hz, 1H, C-5 H), 3.46 (s, 3H, OCH₃), 2.76 (bd, J = 6.2 Hz, 1H, OH), 1.92 (bt, J = 4.4 Hz, 1H, OH). ¹³C NMR (CDCl₃, 50 MHz): $\delta = 139.8$ (C-2), 120.2 (C-3), 115.3 (CN), 94.2 (C-1), 70.5 (C-4), 62.7 (C-5), 61.9 (C-6), 56.3 (OCH₃). HRMS: M⁺-OCH₃ m/e obs.: 154,0511 (C₇H₈O₃N m/e calc.: 154,0506). LRMS (E.I.): m/e (rel. int.): 154 (20), 136 (7), 125 (100), 110 (11), 94 (18), 82 (8), 60 (9), 54 (8), 45 (11).

Methyl 3-cyano-2,3-dideoxy-4,6-O-ditert-butyl-dimethylsilyl- α -D-erythro-hex-2-enopyranoside (**11**): colorless oil. IR (KBr): ν_{\max} (cm⁻¹) = 2940, 2895, 2240 (CN), 1460, 1370, 1265, 1105, 990, 890, 850, 790. ¹H NMR (CDCl₃, 200 MHz): $\delta = 6.45$ (dd, J₁ = 2.9 Hz, J₂ = 1.8 Hz, 1H, vinyl), 4.92 (dd, J₁ = 2.9 Hz, J₂ = 1.5 Hz, 1H, anomeric), 4.29 (dt, J₁ = 8.8 Hz, J₂ = 1.5 Hz, 1H, C-4 H), 3.79 (d, J = 3.0 Hz, 2H, C-6 H), 3.75 (dt, J₁ = 8.9 Hz, J₂ = 3.0 Hz, 1H, C-5 H), 3.42 (s, 3H, OCH₃), 0.93 (s, 9H, SiC(CH₃)₃), 0.90 (s, 9H, SiC(CH₃)₃), 0.24 (s, 3H, SiCH₃), 0.14 (s, 3H, SiCH₃), 0.07 (s, 6H, 2 SiCH₃). ¹³C NMR (CDCl₃, 50 MHz): $\delta = 140.1$ (C-olefine), 121.5 (C-olefine), 116.4 (CN), 94.0 (C-1), 72.1 (C-4),

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62.8 (C-5), 61.6 (C-6), 55.8 (OCH₃), 38.5 (SiC(CH₃)₃), 31.0 (SiC(CH₃)₃), 25.7 (6C, SiC(CH₃)₃), 4.38 (SiCH₃), 4.15 (SiCH₃), 4.48 (SiCH₃), 3.06 (SiCH₃). HRMS: M⁺-CH₃ m/e obs.: 398,2193 (C₁₉H₃₆O₄NSi₂ m/e calc.: 398,2187); M⁺-OCH₃ m/e obs.: 382,2219 (C₁₉H₃₆O₃NSi₂ m/e calc.: 382,2237); M⁺-C(CH₃)₃ m/e obs.: 356,1724 (C₁₆H₃₀O₄NSi₂ m/e calc.: 356,1718). LRMS (E.I.): m/e (rel. int.): 382 (11), 356 (95), 324 (15), 240 (54), 182 (11), 149 (16), 117 (100), 89 (28), 73 (92), 59 (13), 41 (16).

Methyl 2,3-dideoxy-3-C-formyl-4,6-O-ditert-butyl-dimethylsilyl- α -D-erythro-hex-2-enopyranoside (**12**): colorless oil. IR (KBr): ν_{\max} (cm⁻¹) = 2925, 2860, 1700 (CO aldehyde), 1465, 1360, 1255, 1122, 1095, 1060, 984, 871, 840, 778. ¹H NMR (CDCl₃, 200 MHz): δ = 9.55 (s, 1H, aldehyde), 6.57 (dd, J₁ = 3.0 Hz, J₂ = 1.1 Hz, 1H, vinyl), 5.08 (dd, J₁ = 3.0 Hz, J₂ = 0.7 Hz, 1H, anomeric), 4.60 (d, J = 7.1 Hz, 1H, C-4 H), 3.80 (m, 3H, C-5 H and C-6 H), 3.47 (s, 3H, OCH₃), 0.89 (s, 9H, SiC(CH₃)₃), 0.86 (s, 9H, SiC(CH₃)₃), 0.15 (s, 3H, SiCH₃), 0.13 (s, 3H, SiCH₃), 0.06 (s, 6H, 2 SiCH₃). ¹³C NMR (CDCl₃, 50 MHz): δ = 191.5 (C-carbonyl), 143.3 (C-3), 140.2 (C-2), 94.5 (C-1), 73.6 (C-4), 62.6 (C-6), 62.2 (C-5), 55.5 (OCH₃), 25.9 (3C, SiC(CH₃)₃), 25.8 (3C, SiC(CH₃)₃), 18.2 (2C, SiC(CH₃)₃), -4.2 (SiCH₃), -4.4 (SiCH₃), -5.3 (SiCH₃), -5.5 (SiCH₃).

Methyl 3-cyano-2,3-dideoxy-4,6-O-acetate- α -D-erythro-hex-2-enopyranoside (**13**): colorless oil. IR (KBr): ν_{\max} (cm⁻¹) = 2950, 2230 (CN), 1755 (CO acetates), 1448, 1372, 1240, 1134, 1058, 976. ¹H NMR (CDCl₃, 200 MHz): δ = 6.58 (dd, J₁ = 2.5 Hz, J₂ = 1.3 Hz, 1H, vinyl), 5.58 (bd, J = 9.4 Hz, 1 H, C-4 H), 5.05 (bd, J = 2.5 Hz, 1H, anomeric), 4.25-4.05 (complex m, C-5 H, C-6 H_{ax} and C-6 H_{eq}), 3.47 (s, 3H, OCH₃), 2.18 (s, 3H, OC(O)CH₃), 2.10 (s, 3H, OC(O)CH₃). ¹³C NMR (CDCl₃, 50 MHz): δ = 170.4 (C-carbonyl), 169.4 (C-carbonyl), 141.2 (C-2), 116.8 (CN), 114.2 (C-3), 94.2 (C-1), 66.8 (C-4), 62.5 (C-5), 62.0 (C-6), 56.4 (OCH₃), 20.5 (OC(O)CH₃), 20.4 (OC(O)CH₃).

Cycloadduct **2**: colorless oil. $[\alpha]_D^{25}$ = +55.7 (c 1.01, CHCl₃). IR (film): ν_{\max} (cm⁻¹) = 2990, 2930, 1730 (CO aldehyde), 1465, 1386, 1109, 1072, 1039, 758, 708. ¹H NMR (CDCl₃, 200 MHz): δ = 9.94 (s, 1H, aldehyde), 7.45-7.34 (m, 5H, aromatic), 6.43 (bs, 2H, vinyl), 5.41 (s, 1H, benzylic), 4.34 (d, J = 3.7 Hz, 1H, anomeric), 4.27 (dd, J₁ = 10.4 Hz, J₂ = 5.0 Hz, 1H, C-6 H_{eq}), 3.81 (m, 1H, C-5 H), 3.53 (t, J = 10.4 Hz, 1H, C-6 H_{ax}), 3.51 (d, J = 10.4 Hz, 1H, C-4 H), 3.41 (bs, 1H, C-16 H), 3.36 (s, 3H, OCH₃), 3.00 (bs, 1H, C-14 H), 2.78 (t, J = 3.7 Hz, 1H, C-2 H), 1.50 (dt, J₁ = 9.0 Hz, J₂ = 1.7 Hz, 1H, C-15 H), 1.21 (d, J = 9.1 Hz, 1H, C-15 H). ¹³C NMR (CDCl₃, 50 MHz): δ = 201.8 (C formyl), 138.1 (C olefine), 137.0 (C-8), 136.8 (C-olefine), 129.1 (C-11), 128.2 (2C, aromatic), 126.1 (2C, aromatic), 101.9 (C-7), 101.8 (C-1), 80.4 (C-4), 69.6 (C-6), 60.9 (C-5), 60.8 (C-3), 55.0 (OCH₃), 47.0 (C-15), 46.9 (C-2), 46.0 (C-14), 45.5 (C-16). HRMS: M⁺

m/e obs.: 342,1467 (C₂₀H₂₂O₅ m/e calc.: 342,1472). LRMS (E.I.): m/e (int. rel.): 159 (57), 122 (27), 105 (100), 85 (53), 69 (68), 55 (37), 43 (45). Elemental analysis: calc. for C₂₀H₂₂O₅: C = 70.16, H = 6.48, O = 23.36. Obs.: C = 70.06, H = 6.52.

Cycloadducts **4** and **5**: IR (film): ν_{\max} (cm⁻¹) = 2930, 2870, 1730 (CO), 1715 (CO), 1465, 1370, 1205, 1105, 1065, 1035, 765, 705. Cycloadduct **4**: ¹H NMR (CDCl₃, 200 MHz): δ = 7.50-7.30 (m, 5H, aromatic), 6.41 (bs, 2H, vinyls), 5.40 (s, 1H, benzylic), 4.32 (d, J = 3.7 Hz, 1H, anomeric), 4.24 (dd, J₁ = 10.4 Hz, J₂ = 5.2 Hz, 1H, C-6 H_{eq}), 3.85 (m, 1H, C-5 H), 3.53 (m, 1H, C-16 H), 3.51 (t, J = 10.2 Hz, 1H, C-6 H_{ax}), 3.39 (d, J = 7.4 Hz, 1H, C-4 H), 3.38 (s, 3H, OCH₃), 2.95 (m, 1H, C-14 H), 2.80 (t, J = 3.7 Hz, 1H, C-2 H), 2.37 (s, 3H, CH₃), 1.56 (dt, J₁ = 8.9 Hz, J₂ = 1.8 Hz, 1H, C-15 H), 1.12 (bd, J = 8.9 Hz, 1H, C-15 H). ¹³C NMR (CDCl₃, 50 MHz): δ = 209.0 (C carbonyl), 138.0 (C olefine), 137.0 (C-olefine), 135.0 (C-8), 129.0 (C-11), 128.2 (2C, aromatic), 126.1 (2C, aromatic), 102.2 (C-7), 102.0 (C-1), 80.4 (C-4), 69.9 (C-6), 63.6 (C-3), 59.8 (C-5), 55.0 (OCH₃), 49.6* (C-2), 48.0* (C-15), 47.6* (C-16), 46.0 (C-14), 29.4 (CH₃). Cycloadduct **5**: ¹H NMR (CDCl₃, 200 MHz): δ = 7.50-7.30 (m, 5H, aromatic), 6.15 (m, 2H, vinyl), 5.49 (s, 1H, benzylic), 4.60 (d, J = 3.2 Hz, 1H, anomeric), 4.24 (dd, J₁ = 10.4 Hz, J₂ = 5.2 Hz, 1H, C-6 H_{eq}), 3.85 (m, 1H, C-5 H), 3.65 (d, J = 8.8 Hz, 1H, C-4 H), 3.53 (t, J = 10.2 Hz, 1H, C-6 H_{ax}), 3.41 (s, 3H, OCH₃), 3.24 (bs, 1H, C-16 H), 2.69 (bs, 1H, C-14 H), 2.27 (t, J = 3.2 Hz, 1H, C-2 H), 2.17 (s, 3H, CH₃), 1.72 (d, J = 8.9 Hz, 1H, C-15 H), 0.87 (d, J = 8.9 Hz, 1H, C-15 H). ¹³C NMR (CDCl₃, 50 MHz): δ = 205.0 (C carbonyl), 137.8 (C olefine), 137.3 (C-olefine), 135.0 (C-8), 129.0 (C-11), 128.2 (2C, aromatic), 126.1 (2C, aromatic), 103.8 (C-1), 102.2 (C-7), 82.1 (C-4), 69.9 (C-6), 63.6 (C-3), 60.1 (C-5), 55.0 (OCH₃), 47.9* (C-2), 47.8* (C-15), 47.2* (C-16), 46.0 (C-14), 30.0 (CH₃).

Cycloadduct **14**: colorless oil. ¹H NMR (CDCl₃, 200 MHz): δ = 9.90 (s, 1H, aldehyde), 6.31 (m, 2H, vinyl), 4.31 (d, J = 5.2 Hz, 1H, anomeric), 3.85-3.60 (m, 4H, C-4 H, C-5 H, C-6 H), 3.38 (s, 3H, OCH₃), 3.35 (bs, 1H, C-9 H), 2.95 (bs, 1H, C-7 H), 2.36 (dd, J₁ = 5.2 Hz, J₂ = 3.7 Hz, 1H, C-2 H), 1.5 (dt, J₁ = 8.9 Hz, J₂ = 1.7 Hz, 1H, C-8 H), 1.3 (bd, J = 9.0 Hz, 1H, C-8 H), 0.91 (s, 9H, SiC(CH₃)₃), 0.87 (s, 9H, SiC(CH₃)₃), 0.17 (s, 3H, SiCH₃), 0.07 (s, 3H, SiCH₃), 0.06 (s, 6H, 2 SiCH₃). ¹³C NMR (CDCl₃, 50 MHz): δ = 202.3 (C formyl), 137.9 (C-olefine), 136.3 (C-olefine), 101.3 (C-1), 73.2 (C-4), 69.3 (C-5), 62.7 (C-6), 61.8 (C-3), 55.0 (OCH₃), 49.3* (C-7), 47.7 (C-8), 45.9* (C-9), 45.5 (C-2), 25.8 (3C, SiC(CH₃)₃), 25.7 (3C, SiC(CH₃)₃), 18.1 (2C, SiC(CH₃)₃), -4.05 (SiCH₃), -4.70 (SiCH₃), -5.42 (SiCH₃), -5.62 (SiCH₃).

Cycloadduct **15**: ¹H NMR (CDCl₃, 200 MHz): δ = 9.61 (s, 1H, aldehyde), 6.15 (m, 2H, vinyl), 4.61 (d, J = 4.5 Hz, 1H, anomeric), 4.02 (d, J = 9.8 Hz, 1H, C-4 H), 3.80-3.65

(m, 3H, C-5 H, C-6 H), 3.42 (s, 3H, OCH₃), 3.17 (bs, 1H, C-9 H), 2.68 (bs, 1H, C-7 H), 1.94 (dd, J₁ = 4.4 Hz, J₂ = 1.9 Hz, 1H, C-2 H), 1.5 (d, J = 8.9 Hz, 1H, C-8 H), 1.3 (d, J = 9.0 Hz, 1H, C-8 H), 0.91 (s, 9H, SiC(CH₃)₃), 0.87 (s, 9H, SiC(CH₃)₃), 0.12 (s, 3H, SiCH₃), 0.07 (s, 6H, 2 SiCH₃), 0.06 (s, 3H, SiCH₃).

Cycloadduct **17**: colorless oil. IR (KBr): ν_{\max} (cm⁻¹) = 2942, 2248 (CN), 1761 (CO acetates), 1456, 1379, 1249, 1227, 1154, 1103, 1058, 1026. ¹H RMN (CDCl₃, 200 MHz): δ = 6.38 (m, 2H, vinyls), 4.56 (d, J = 9.9 Hz, 1H, C-4 H), 4.29 (d, J = 4.64 Hz, 1H, anomeric), 4.26-4.05

(complex, 3H, C-5 H, C-6 H_{ax} and C-6 H_{eq}), 3.37 (s, 3H, OCH₃), 3.28 (m, 1H, C-9 H), 3.08 (m, 1H, C-7 H), 2.83 (dd, J₁ = 4.6 Hz, J₂ = 3.4 Hz, 1H, C-2 H), 2.17 (s, 3H, OC(O)CH₃), 2.06 (s, 3H, OC(O)CH₃), 1.74 (m, 1H, C-8 H), 1.71 (m, 1H, C-8 H). ¹³C RMN (CDCl₃, 50 MHz): δ = 170.4 (C carbonyl), 169.7 (C carbonyl), 137.1 (C olefine), 136.2 (C olefine), 121.6 (CN), 100.5 (C-1), 67.4 (C-4), 67.3 (C-5), 62.7 (C-6), 55.4 (OCH₃), 54.2 (C-9), 52.0 (C-7), 48.0 (C-8), 47.3 (C-3), 45.7 (C-2), 20.7 (2C, OC(O)CH₃).
